VOL. LV, NO. 12, DECEMBER, 1960

THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

PUBLISHED MONTHLY BY
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PUBLICATION OFFICE, EASTON, PA.

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The Journal

of the

AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. LV DECEMBER 1960 No. 12

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"When we first saw the mellowness, roundness, and overall high quality of this kidskin – 29 years ago – we knew what Zircotan could do."

Dr. Ian Somerville, right, and Mr. Richard Fisher of Rohm & Haas are discussing the early days of zirconium tanning materials.

Dr. Somerville himself supervised tanning, in a commercial tannery, the piece of kidskin shown here. Though this was 29 years ago, the leather is still round, plump, and mellow.

Rohm & Haas first supplied zirconium tanning materials commercially as acidic solutions. Continuing development work then produced in 1938 a dry acid salt much easier to handle and use.

Over the past 20 years, it is estimated that more than 75 million square feet of leather have been made in the U.S. using this salt, now known by the trade name, ZIRCOTAN.

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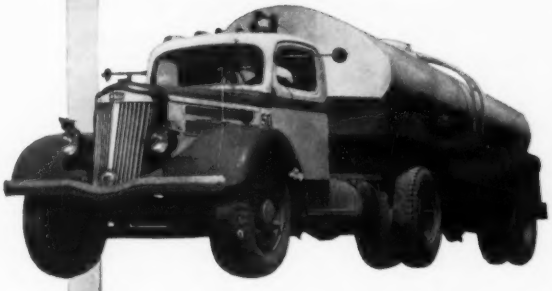

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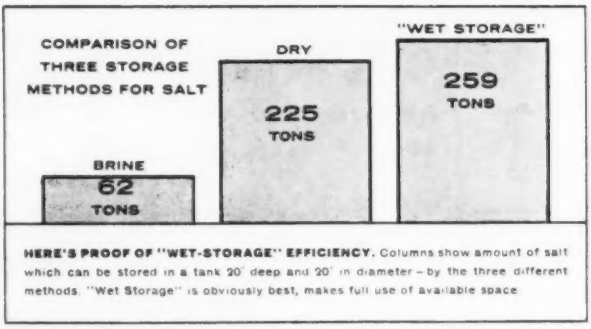

HERE'S PROOF OF "WET-STORAGE" EFFICIENCY. Columns show amount of salt which can be stored in a tank 20' deep and 20' in diameter – by the three different methods. "Wet Storage" is obviously best, makes full use of available space.

A problem frequently encountered in the leather-tanning industry is how to make the best use of available tank space for rock-salt storage. There are three basic ways to store salt in tanks. Each one has certain advantages.

1. Salt stored in the form of fully saturated brine offers the important advantage of convenience: the brine can be *pumped* to points of use in the plant, eliminating salt-handling expense. However, the amount of salt which can be stored in this way is relatively small, because each cubic foot of brine contains only about 19¾ lbs. of salt in dissolved form.

2. Salt stored in dry form makes far better use of available tank space: each cubic foot contains an average of 72 lbs. of rock salt—about 3.6 times the amount which can be stored as brine. However, moving dry salt to points of use is time-consuming, inconvenient and expensive. And dry-salt measurement is not as exact, or as easy, as measurement of fully saturated brine.

3. "Wet Storage"—which actually combines features of dry storage and brine storage—gives the benefits of fully saturated brine, along with the space-saving storage of dry salt. Here's the story:

- With "Wet Storage", any given tank can store *more* salt than is possible when dry salt or saturated brine are stored by themselves.
- There is a constantly available supply of fully saturated brine for every plant need.
- Much less handling of rock salt is needed. There are fewer salt deliveries, fewer brine-making problems.

How "Wet Storage" works. When a tank is filled with dry rock salt, almost half the storage space is actually "empty," because the salt crystals don't pack solidly together. But when the voids between the crystals are occupied by fully saturated brine, additional amounts of salt can be stored. This is because fully saturated brine itself contains 2.65 lbs. of salt per gallon.

Lixator best example of "Wet Storage" principle. Storing salt under covering brine is the basic operating principle of International's exclusive Lixate Brine-Making Process. In the Lixator, rock salt and fully saturated brine are combined, making fullest use of available storage space. The Lixator provides pure, crystal-clear brine for immediate use when and where it's needed.

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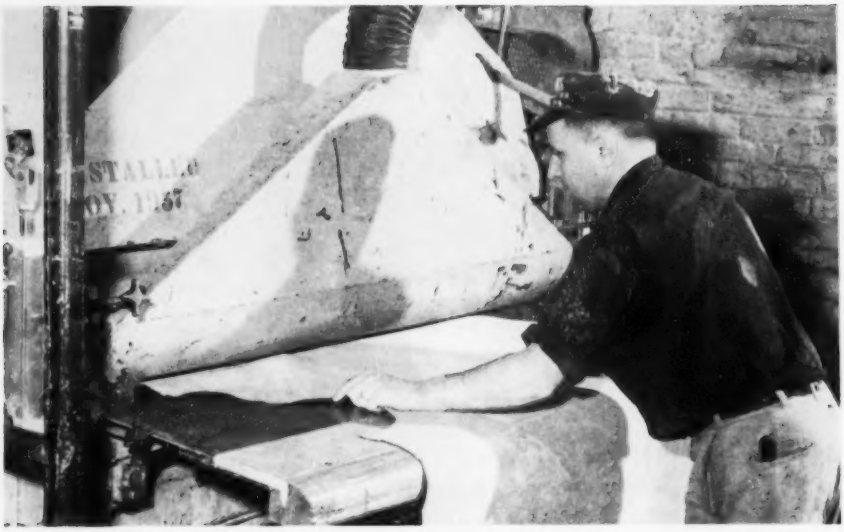

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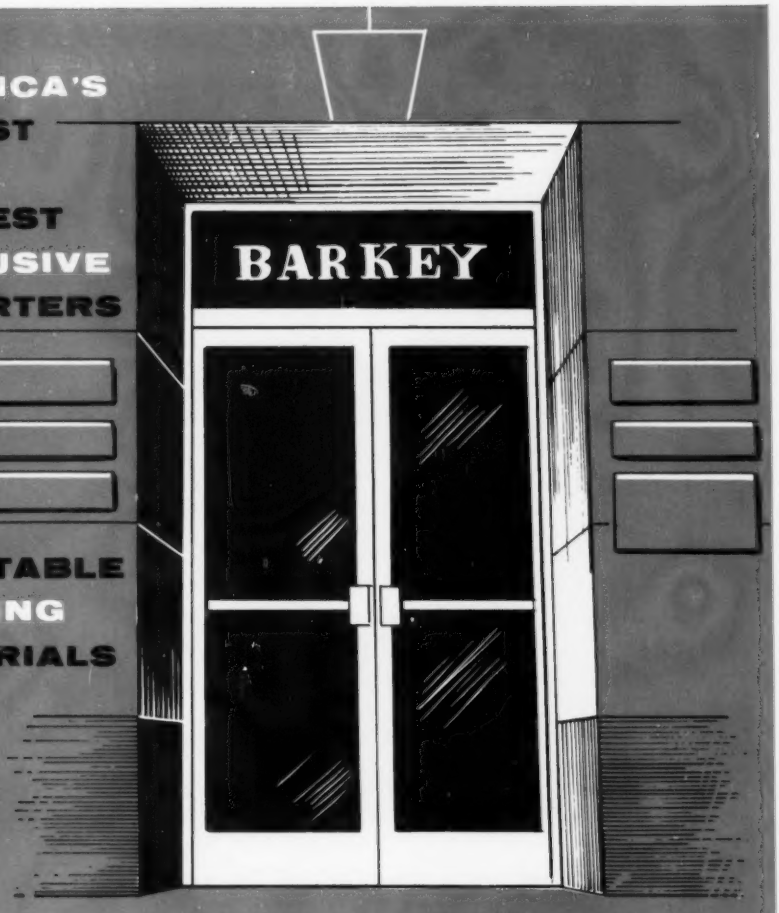

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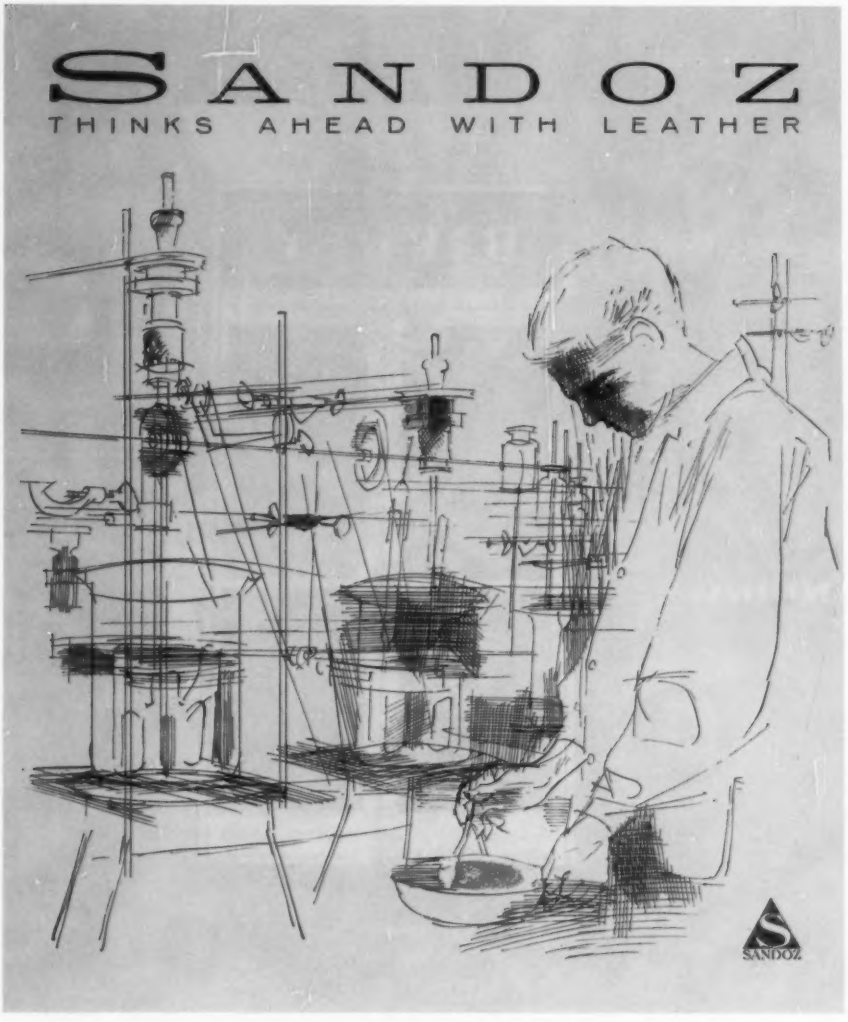

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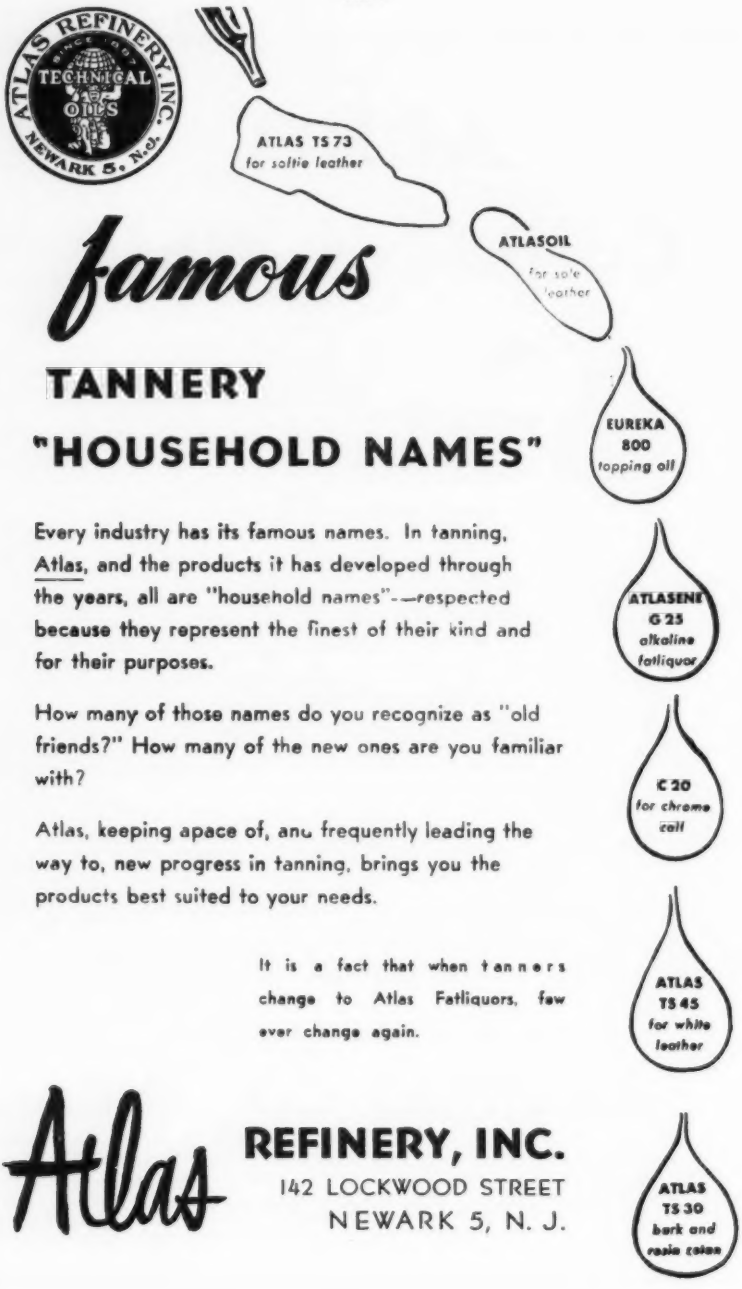

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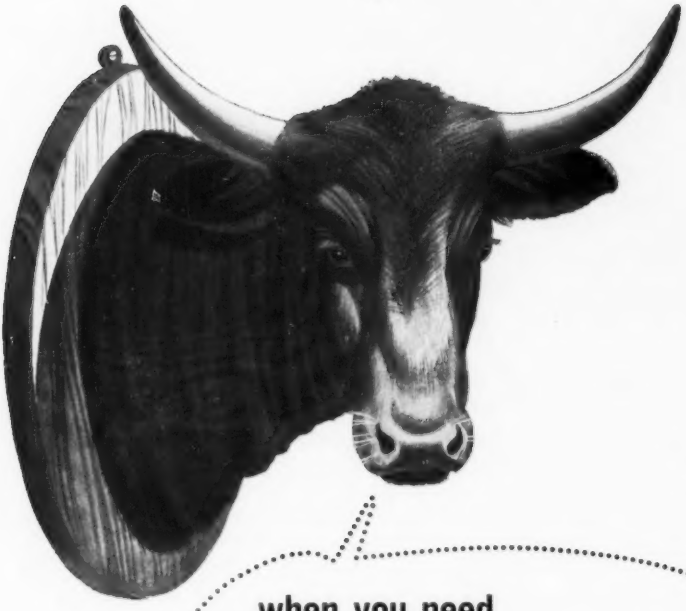

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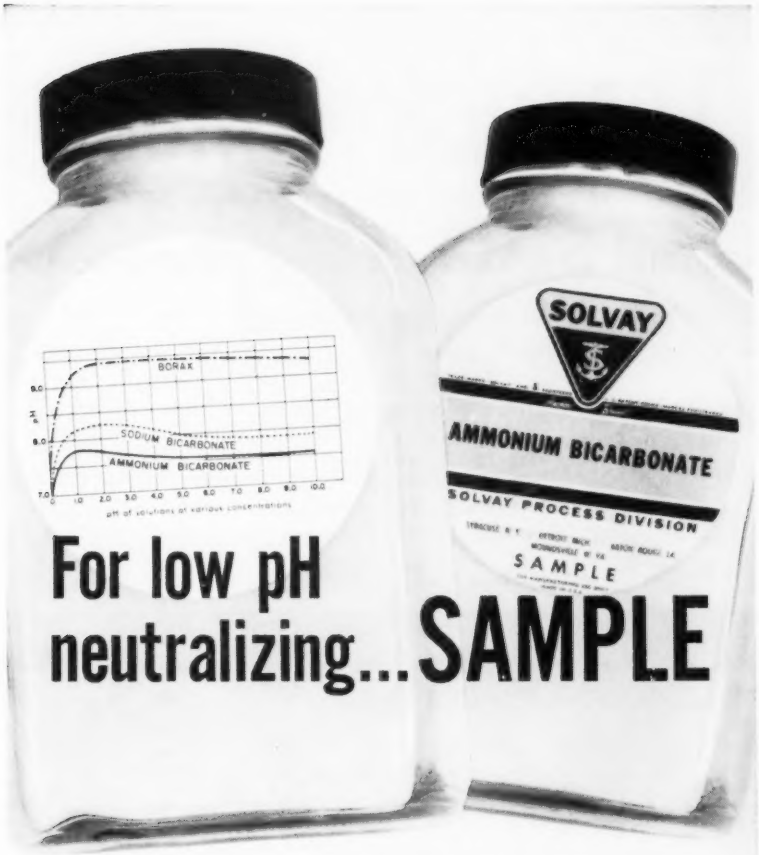

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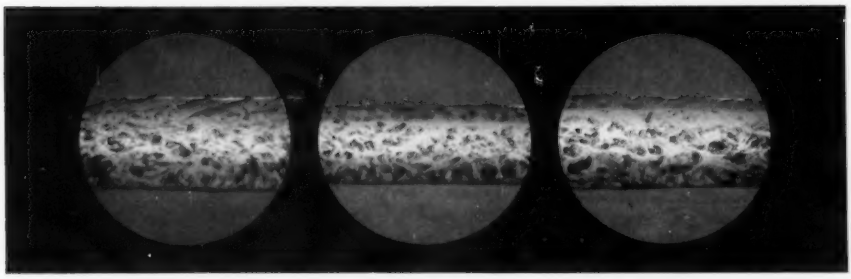

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ZIRCONIUM TANNAGE. VIII

THE STRATIGRAPHIC DISTRIBUTION OF ZIRCONIUM IN LEATHER*

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ABSTRACT

A problem in practical tanning utilizing insoluble sodium zirconium silicate as described previously led to work on the distribution of zirconium and silica throughout the skin. This culminated in stratigraphic analyses of unsplit cowhide leathers tanned with a number of commercially available zirconium salts in comparison with chrome. These results are reported, and some significant aspects are discussed.

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INTRODUCTION

Three years ago a new approach to zirconium tannage was announced (1). This consisted in the direct utilization of insoluble zirconium derivatives, and the one selected for development work was sodium zirconium silicate. We reported that this could be reacted with dilute acid in the tanning drum in the presence of pickled skins, which were converted to leather by the basic zirconium sulfate produced in the reaction. At the same time silica was liberated in the form of water-soluble silicic acid which was taken up by the skins and contributed materially to the filling effect characteristic of zirconium tannage.

The method of adding sodium zirconium silicate directly to the skins in the tanning liquor in conjunction with the large amount of sulfuric acid required to form basic zirconium sulfate was a radical one, hence its development on a tannery scale was begun cautiously. In practice it has been found that this procedure is quite feasible both in making white leather from pickled stock and in the retannage of chrome leather. Many thousands of feet of satisfactory leather have been produced with a minimum amount of trouble;

*Presented by Dr. Somerville on June 16, 1959, at the Annual Meeting of The American Leather Chemists Association, Mackinac Island, Michigan.

in fact, where the recommended procedures were followed the acidification step has taken place smoothly and without causing any damage whatever to hides or skins. However, the additional care required in operating this process apparently was not compensated for sufficiently by the reduced cost. Recently a new form of basic zirconium sulfate containing silica has become available in this country, a form utilizing the complementary action of these tanning materials more simply and economically. Our development work to date has indicated that this product, designated Zircotan T, can replace the process of acidification of sodium zirconium silicate as well as the conventional tannage with the more expensive sodium zirconium sulfate.

In the course of a small-scale tannery experiment using sodium zirconium silicate plus acid, some unexpected defects showed up for which no reason could be found by conventional analysis. This led us to apply stratigraphic methods to determine the distribution of the mineral components in the leather. While the results did not point to any one factor as responsible for the trouble described, they did suggest that it may have been caused by a combination of several specific factors. Apart from this minor production problem the analyses revealed some interesting and important facts about the penetration of zirconium and silica into leather. This led us to study the whole question of their distribution when tanning with a series of commercial zirconium salts.

DISTRIBUTION OF ZIRCONIUM IN LEATHER

Our first consideration of this arose in the course of a small-scale tannery experiment in which unsplit cowhides were tanned using sodium zirconium silicate in conjunction with acid. An excellent result was obtained using the recommended procedures for straight mineral tannage, but some difficulty was experienced on a few sides retanned with a phenolic syntan. A small percentage of oxalic acid had been given along with the acidic syntan to minimize the chance of stains from iron, and a good color was obtained. However, when these sides were dried out rather rapidly with warm air at low humidity, the grain showed some weakness, although cuttings dried at room temperature were entirely satisfactory, as were the sides dried rapidly without retan. This result was surprising because of the peculiar incidence of the trouble and because nothing like this had been observed before in many years of experience with zirconium tannage. Analyses were run by standard procedures which have been described previously (2), and the results are given in Table I. The figures are typical of this type of tannage and showed no marked difference between these leathers. Accordingly, a grain split 0.05″ in thickness was made from both leathers using a Lab Model 6″ Landis splitting machine. The analytical figures were still quite similar but showed lower fixation of zirconium in the weak grain. A thinner grain

TABLE I

TANNAGE OF UNSPLIT COWHIDE

OVER-ALL ANALYSIS—0.25" THICK

	Zircotan S Alone	Zircotan S Retanned with Syntan + Oxalic Acid
	Air-dry basis (%)	
ZrO_2	12.4	12.2
SiO_2	5.7	5.9
TiO_2	1.1	2.3
Oil (by $CHCl_3$)	8.3	7.1
Hide substance	56.6	56.0
	Hide-substance basis (%)	
ZrO_2	21.9	21.8
SiO_2	10.1	10.6
TiO_2	1.9	4.1
Oil (by $CHCl_3$)	14.7	12.7
Total Oxides	33.9	36.5
$\frac{SiO_2 + TiO_2}{ZrO_2}$	0.55	0.67

TABLE II

TANNAGE OF UNSPLIT COWHIDE

ANALYSIS OF GRAIN LAYER—0.03" THICK

	Zircotan S Alone	Zircotan S Retanned with Syntan + Oxalic Acid
	Air-dry basis (%)	
ZrO_2	11.3	7.3
SiO_2	10.0	10.4
TiO_2	5.3	8.7
Oil (by $CHCl_3$)	26.0	22.0
Hide substance	28.1	32.4
	Hide-substance basis (%)	
ZrO_2	40.2	22.5
SiO_2	35.5	32.0
TiO_2	18.7	26.7
Oil (by $CHCl_3$)	92.0	67.8
Total Oxides	94.4	81.2
$\frac{SiO_2 + TiO_2}{ZrO_2}$	1.3	2.6

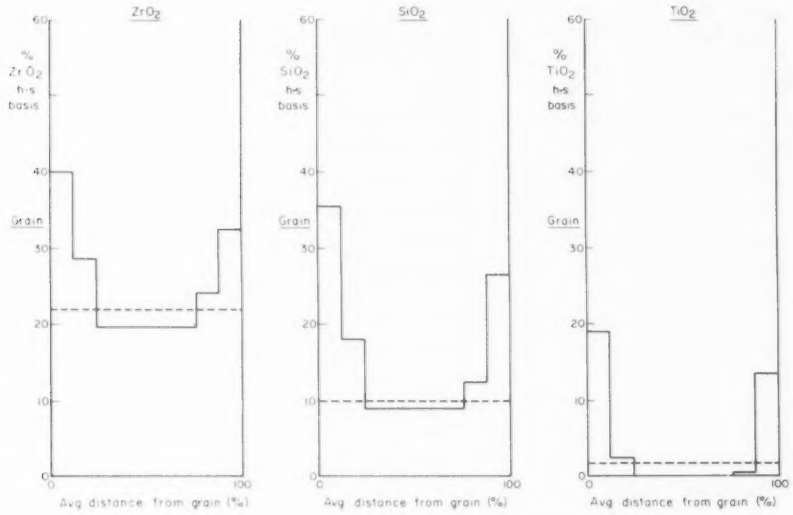

FIGURE 1.—Zircotan S alone. Tannery experiments.
---------- Over-all analysis.

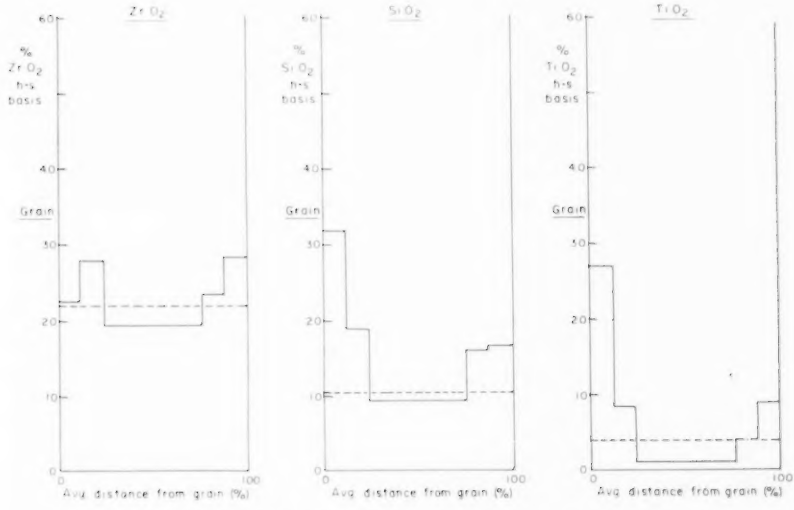

FIGURE 2.—Zircotan S. Syntan retan. Tannery experiments.
---------- Over-all analysis.

split 0.03″ in thickness (about one-eighth of the total) was then made from both leathers, and the results are given in Table II. The straight mineral-tanned leather was heavily loaded on the grain, yet showed no weakness, whereas that given the retan seemed more normal in composition, yet was tender. It did, however, have considerably less surface oil and a much higher proportion of silica plus titanium oxide relative to the zirconium oxide present.

In order to get a more complete picture it was decided to run stratigraphic analyses on these two leathers. Another layer approximately 0.03″ thick was split from the same samples under the original grain, and also two corresponding layers from the flesh, leaving the center intact and about 0.12″ in thickness. Analyses were run by the same method on these layers. The results for the straight mineral tannage are shown in Fig. 1, in which the grain of the leather is at the left, and each strip in turn, going toward the flesh, shows an average value throughout its thickness as indicated. The extent of surface loading with all mineral components shows up very markedly when contrasted with the figures obtained for over-all analysis shown by the dotted lines. The corresponding results for the syntan retan are shown in Fig. 2, and while the same general pattern is obtained, there is, as pointed out above, less ZrO_2 in the surface but a much higher ratio of (SiO_2 plus TiO_2) to ZrO_2.

We studied on a laboratory scale the various factors involved in this tannery experiment but were not able to reproduce exactly the detrimental effects described. Evidence was obtained, however, that this weakness must have been caused by a combination of circumstances, none of which by itself could be held responsible. These included excessive loading of the grain surface, particularly with TiO_2; the addition of oxalic acid along with the acidic syntan; lower fixation of oil in the grain; and drying conditions which were too drastic for this modification of the tannage although entirely satisfactory for the corresponding sides with a straight zirconium tannage.

Other implications of these results are of far greater importance than the immediate problem which led to this work. For example, let us contrast the relative distribution of zirconium, silica, and titanium. In the center of the hide the amount of zirconium is a little less than 20%, while at the same time the silica amounts to just under 10% on a hide-substance basis. This ratio of SiO_2/ZrO_2 is 1.0 on a molar basis, the same as in the original tanning material, confirming that the silicic acid liberated by acid along with basic zirconium sulfate penetrates the hide in the same way as the latter. On the other hand, as one might suspect, the TiO_2 behaves as an inert pigment, and very little penetrates to the center of the skin, most being located on the extreme surfaces. This contrast suggested the application of the same method to study the distribution of mineral components when tanning with soluble and insoluble silica in conjunction with basic zirconium sulfate.

STRATIGRAPHIC DISTRIBUTION WITH SOME COMMERCIAL TANNING SALTS

It was decided to run a series of tests on strips of unsplit cowhide, comparing the stratigraphic distribution of the mineral constituents when tanning with a basic chrome sulfate, a basic zirconium sulfate, sodium zirconium silicate acidified in the tanning liquor, and two commercial samples of basic zirconium sulfate containing silica. In each case a comparison was run between adding the tanning material directly to the pickled strip and first giving a pretreatment with 1% citric acid. In addition, half of each zirconium leather was retanned with 10% of a high-quality phenolic syntan showing 15% tans by analysis. A summary of the tests is shown in Table III, while the method of processing was briefly as follows:

TABLE III

Pretreatment	Mineral Tan	Composition
2% sodium formate	10% Tanolin R	chromic sulfate, 36% basicity
1% citric acid 2% sodium formate	10% Tanolin R	
None	25% Zircotan N	sodium zirconium sulfate, 45% basicity
1% citric acid	" " "	*JALCA*, **38**, 326 (1948)
None	18% Zircotan S	sodium zirconium silicate
1% citric acid	" " "	*JALCA*, **51**, 553 (1956)
None	30% Zircotan T	sodium zirconium sulfate, containing
1% citric acid	" " "	silica; 36% basicity
None	35% ZT-45	sodium zirconium sulfate, containing
1% citric acid	" " "	insoluble silica; 45% basicity

Corresponding pieces were given 10% Syntan retan (Leukanol C).

A commercially processed unsplit cowside in the pickled condition was cut into strips running from backbone to belly. The pieces to be tanned with chrome initially showed pH 3.2 and were given the equivalent of 2.5% Cr_2O_3 in 200% of a 5% salt solution. They were neutralized in the tan liquor with sodium bicarbonate to pH 3.8, washed, and fatliquored. The strips for zirconium tannage were drummed in 200% of a 5% salt solution, given additional sulfuric acid to show pH 1.2, and then the zirconium salt was added dry in an amount equivalent to 8% ZrO_2 (along with the required amount of acid in the case of sodium zirconium silicate). Drumming was continued until penetration was complete, as indicated by a minimum shrinkage temperature of 190° F., then the strips were neutralized in the tanning liquor with sodium bicarbonate to pH 3.5. After each strip was divided

lengthwise, one half was neutralized in a fresh liquor using 2% sodium acetate plus 1% sodium citrate to pH 3.8–4.0, rinsed, and fatliquored with 4% of a sulfonated oil plus 1% raw neatsfoot; the other half was given the syntan retan, neutralized to the same pH using 2% sodium acetate plus 1% sodium oxalate, and fatliquored in the same way.

After the strips were worked up from the crust by dipping, sammying, staking, and tacking, they were split into five layers at Lehigh University through the kindness of Dr. R. Stubbings. The two outside layers from flesh and grain were each around 0.02″ (from 0.013″ to 0.025″) in thickness, while the residual center layer ranged from about 0.06″ to 0.15″. Compensation for local variations in the thickness of the skin is made by Dr. Stubbings on a weight basis, but we adhered to the percentage of average thickness in our comparisons. Actually, very similar results on the distribution of mineral constituents are obtained either way. The importance of this method lies in the correctness of the over-all picture which it gives rather than in the absolute values obtained in one particular area. For example, in this method of operation it is not possible to make any allowance for the specific position on the hide, nor to select analytical samples from official locations.

The actual method of analysis used was that referred to above (2). Initially we decided to simplify the procedure for mineral components by ashing at 600° C., fusing, dissolving in hydrochloric acid and making to volume, determining combined oxides by precipitation with ammonia, driving off the SiO_2 with sulfuric acid and hydrofluoric acid, then calculating ZrO_2 by difference. In a few instances this seemed to give high results in comparison with the values obtained by direct precipitation with *p*-hydroxyphenyl arsonic acid. The latter method has proven very reliable over a long term of years and was therefore adopted for this series of experiments. Silica was then calculated as combined oxides less ZrO_2, although this might include minor amounts of other impurities. The diagrams which follow illustrate some interesting features of the results obtained.

DISTRIBUTION OF MINERAL COMPONENTS

Figure 3 shows the distribution of chrome in heavy cowhide leather tanned in this way. The chrome penetrated readily into the center of the hide, but appreciably more was fixed in the surface layers. There is a very small effect from the citric acid pretreatment, shown by the dotted line. This tends to lower fixation of chrome. The syntan retan had no appreciable effect on the chrome distribution in either case and is not shown here.

Figure 4 shows the results obtained using basic zirconium sulfate (Zircotan N). The zirconium also penetrated readily into the center of the hide and

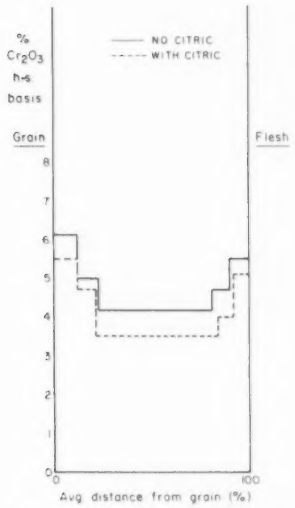

FIGURE 3.—Basic chrome sulfate.

was taken up more efficiently than with chrome. The ratio of ZrO_2/Cr_2O_3 given was approximately 3 to 1, but the ratio found in the center of the hide is over 5 to 1. A notable feature is the high percentage of ZrO_2 in the extreme surface layers found in our initial experiments and confirmed here. We believe that this is a contributory factor to the tight strong grain observed in zirconium tannage. The small amount of SiO_2 found is due to its presence as an impurity in the technical basic zirconium sulfate. The effect of citric acid is again shown by the dotted line and is more pronounced here than in the case of chrome. It reduces the deposition of zirconium on the surface but has a smaller effect on its fixation in the center of the hide. The syntan retan had only a minor effect on the distribution of zirconium and silica and is not shown here.

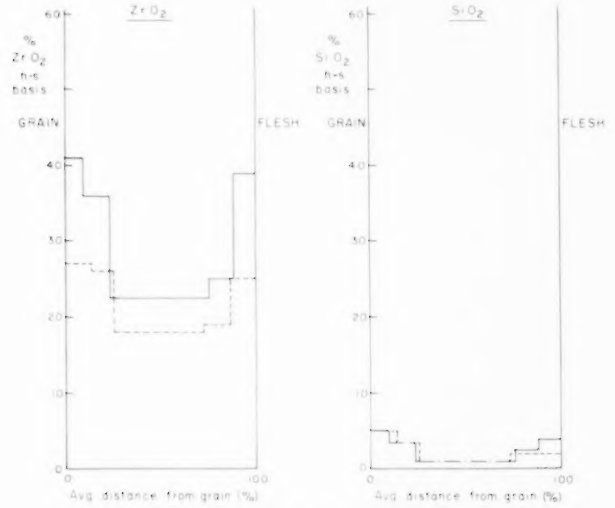

FIGURE 4.—Basic zirconium sulfate.

———— No citric
- - - - - - With citric

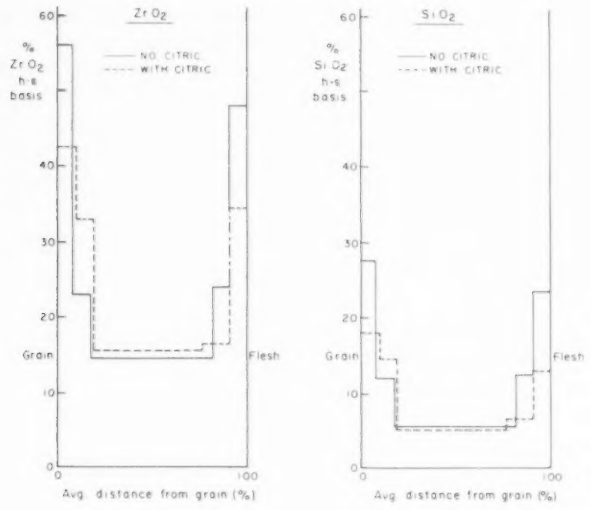

FIGURE 5.—Sodium zirconium silicate, acidified.

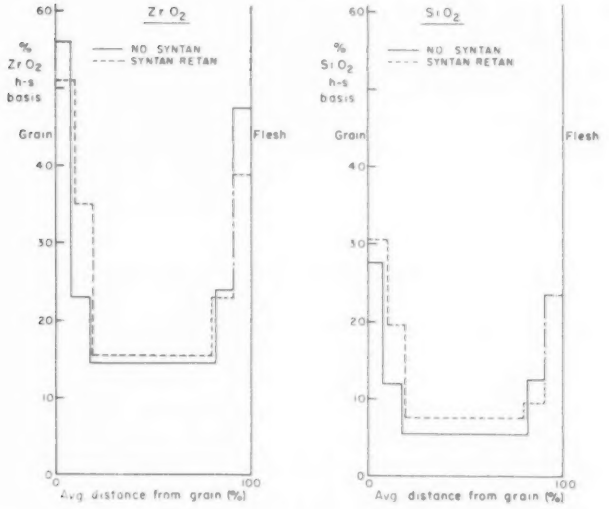

FIGURE 6.—Effect of syntan retan following sodium zirconium silicate, acidified.

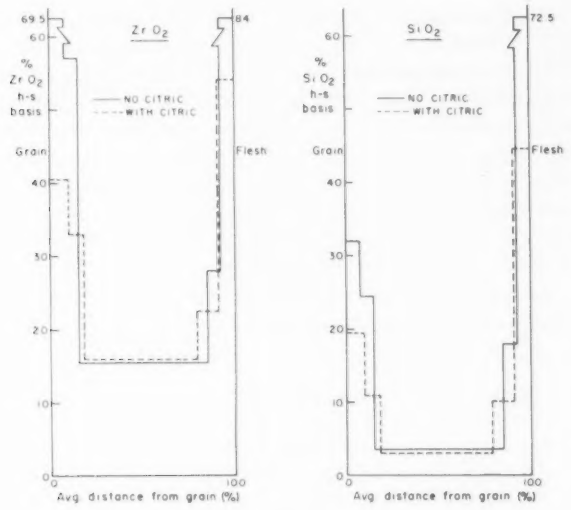

FIGURE 7.—Sodium zirconium sulfate containing silica.

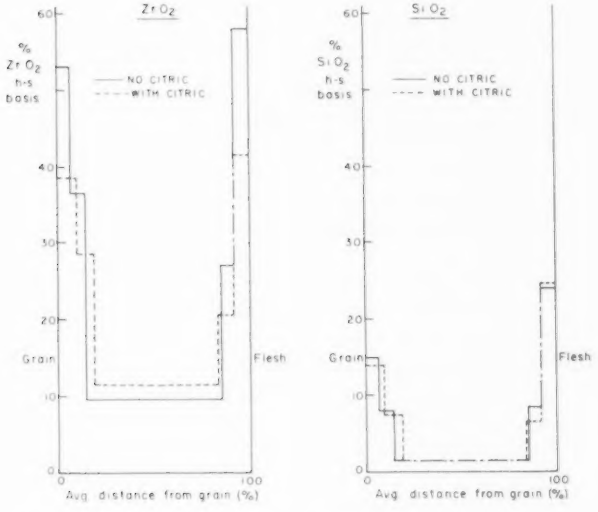

FIGURE 8.—Sodium zirconium sulfate containing insoluble silica.

Figure 5 shows the corresponding results for sodium zirconium silicate (Zircotan S). In this case the penetration of zirconium is also very good, though the fixation in the center is lower than in the absence of silica. There is a higher percentage of ZrO_2 in the surface layers than with sodium zirconium sulfate. The effect of citric acid is again pronounced, giving lower deposition of zirconium on the surface and tending to equalize its distribution throughout the hide. It is of interest to note the behavior of silica in this experiment. The molecular ratio for SiO_2 ZrO_2 in the original product is 1.0, while the ratio found here in the center of the hide is about 0.8 without citric acid and about 0.7 with 1% citric, a high ratio of silica to zirconium. The effect of the syntan retan is shown by the dotted line on Fig. 6. It has only a small effect in the same direction as a citric acid pretreatment but also tends to give more even distribution of zirconium.

Figure 7 shows the corresponding results for sodium zirconium sulfate containing silica (Zircotan T). This product was introduced less than a year ago as an alternative to the acidification of sodium zirconium silicate. In practice it has been found to give rather similar results and to be much simpler in tannery application. These curves show that the penetration of zirconium with this product is almost identical to the previous case, but when used alone, there is a greater build-up on the surface. This was largely overcome on the grain by giving 1% citric acid, but high values were still obtained on the flesh. The silica figures are of interest in that they are almost the same as in the previous case on the grain, lower but still considerable in the center, and high on the flesh. This extent of penetration corresponds to a molecular ratio for SiO_2 ZrO_2 of 0.40–0.45 in the center of the hide.

Some of these figures seem unusually high when calculated on a hide-substance basis, but so far all the leather tanned with this material has been of excellent quality. If we turn to the terminology of the vegetable tanner whose "degree of tannage" is simply combined tannin calculated on a hide-substance basis, we begin to approach, in some of these surface layers of mineral-tanned leather, values for degree of tannage comparable to those of vegetable-tanned leather. It has recently been suggested by Ranganathan and Reed (3) that zirconium appears to have more in common with vegetable tannage than with chrome. This comparison is of value when considering the amount of tanning material present in relation to hide substance, but it may be misleading in terms of the characteristics of the leather produced. For example, the reactivity of zirconium leathers tanned as outlined in this paper toward acid, direct, or basic dyestuffs is entirely different from that of vegetable-tanned leathers. These zirconium leathers behave predominantly as cationic compounds with a strong affinity for acid and direct dyes and little mordanting power for basic dyestuffs, whereas vegetable-tanned leathers show exactly the opposite behavior.

Figure 8 shows comparable results for sodium zirconium sulfate containing insoluble silica (ZT-45). This French product has been extensively described in the literature by Portes (4) and others and has been available for some years in Europe. It is of interest to note that under comparable conditions the zirconium penetrates to a lesser extent than with any of our domestic products. The benefit of citric acid in improving surface distribution is still evident. The most obvious difference from either of our domestic products containing silica is the lower amount of both zirconium and silica found in the center of the skin. This seems to indicate less efficient utilization of tan, though the leather quality of the strips was reasonably good. The molecular ratio of SiO_2: ZrO_2 in this area was about 0.25.

TABLE IV

ANALYSIS OF CENTER AREAS OF HIDE STRIPS
USING VARIOUS ZIRCONIUM SALTS

	Basic Sulfate	Silicate Acidified	Sulfate with Silica	Sulfate with Insoluble Silica
	ZrO_2, %, hide-substance basis			
No citric, no retan	22.5	14.4	15.4	9.6
1% citric, no retan	17.8	15.5	15.8	11.4
	SiO_2, %, hide-substance basis			
No citric, no retan	0.9	5.5	3.3	1.7
1% citric, no retan	0.9	5.2	3.1	1.6

Table IV summarizes the distribution of ZrO_2 and SiO_2 in the center of the hide for all four zirconium tannages. It indicates essentially the same good fixation of ZrO_2 in the second and third cases, in an amount intermediate between the first and fourth. The best penetration of silica occurs in the second case, when it is freshly liberated as silicic acid in the tanning bath, but appreciably more is fixed in the third case than in the fourth.

BASICITY OF MINERAL TANNAGE

In addition to determination of the oxides of the individual metals present, analyses were run to determine the basicity of the final leathers. We followed the Provisional ALCA Method for determination of basicity in chrome leather (May 1954). Even though this has been criticized severely, no agreement has yet been reached in this country on an improved method which can be substituted for it. In any event we were interested in relative rather than absolute values for basicity.

The latest revision of Method D-30 is entitled *Sulfate Basicity*, but reference is made to "chromic oxide that is combined with sulfate *or other acid anions*." Hence the latter are normally included in the calculation for basicity except when firmly held in the chrome leather complex. In some of our analyses of the solutions extracted by water the acidity found by titration was actually higher than the SO_4 obtained gravimetrically, because of some organic acids present, but this is compensated for in calculating the results, e.g., (percent combined acid sulfate [Method D-20]) = (percent total sulfate as SO_4 found gravimetrically on extraction with phosphate or ammonia) − (percent sulfate as SO_4 found gravimetrically in aqueous extract) + (percent free acid calc. as SO_4 found by titration in aqueous extract).

It has been pointed out previously (2) that the basicity of zirconium leathers may be expressed using the Schorlemmer system in the same way as for chrome but with substitution of the proper factor. This is indeed incorporated in Method D-30. The figures shown in Table V were obtained in this way

TABLE V

BASICITY OF CHROME AND ZIRCONIUM LEATHERS

(*Schorlemmer Scale*)

	Grain		Center	Flesh	
CHROME					
1% citric, no retan	58	43	41	48	65
ZIRCOTAN N					
1% citric, no retan	86	70	58	62	77
ZIRCOTAN T					
1% citric, no retan	89	74	59	66	88

and illustrate the type of results obtained under the conditions of these experiments.

In all cases there is a considerable decrease in basicity from the surface to the center of the hide, with the zirconium showing, as anticipated, higher values than the chrome tannage.

DISTRIBUTION OF FATLIQUOR

One of the critical factors in the entire development of zirconium tannage has been selection of the proper fatliquor to give the desired leather characteristics. It is apparent from the information presented in this paper why this is a difficult matter. The tendency to build up a high concentration of

ZrO_2 on or near the surface might prevent adequate penetration of oil when a mellow leather is desired, while increasing the percentage given might lead to excessive oil on the surface without a marked improvement in penetration.

Some of the results of our extraction experiments are shown in Table VI.

TABLE VI

DISTRIBUTION OF FATLIQUOR

(Percentage extracted by chloroform on air-dry basis)

Citric	Retan	Grain		Center		Flesh	Calculated Over-all Analysis Weight Basis
CHROME							
0	none	28.6	7.3	3.2	10.9	25.9	9.8
0	syntan	28.9	14.3	1.5	8.7	21.1	8.2
1	none	29.0	8.6	2.8	14.3	27.6	9.6
1	syntan	31.4	16.1	1.8	11.9	27.2	8.7
ZIRCOTAN N							
0	none	16.2	13.6	2.6	8.8	18.5	7.8
0	syntan	8.2	6.1	1.0	5.1	17.7	4.5
1	none	12.6	6.0	0.7	2.8	13.4	4.8
1	syntan	7.5	2.4	0.6	3.4	14.0	4.0
ZIRCOTAN T							
0	none	12.4	11.5	1.8	15.2	23.5	6.2
0	syntan	13.0	12.1	2.8	17.9	23.2	6.7
1	none	5.7	4.5	1.0	6.0	14.2	3.4
1	syntan	9.4	5.1	2.5	9.6	17.3	5.2

The percentages found on an air-dry basis for each layer were used to calculate the over-all analysis for various leathers. Obviously the take-up of fatliquor was better with chrome than with the zirconium tannages, but the differences between the selected variations in one tannage were not, in general, outstanding. Much more pronounced effects were demonstrated when the composition of the component layers was taken into consideration. The extractable oil in the grain layers of the chrome leathers was much higher than in any of the zirconium leathers and was not appreciably affected by the presence of citric acid or syntan in processing. The extractable oil in the grain layers of these zirconium leathers was in general considerably less when citric acid had been given initially, while in the absence of silica it was also reduced by the subsequent syntan retannage. In spite of these lower values the leathers obtained using citric acid prior to tannage or syntan in retannage usually

were mellower than corresponding strips processed directly. Attention should be called to the low percentage of oil found in the center of the hide, even in the case of the chrome leathers.

This does not cover many aspects of fatliquoring zirconium leather, but it does indicate one effective approach to securing reliable information on this subject.

DISCUSSION OF RESULTS

In looking over these stratigraphic results from a broad viewpoint it is obvious why zirconium gives effects quite different from those obtainable by chrome tannage. While we have always known that a higher percentage of mineral tan is applied and taken up by the hide, giving a well filled leather with good solidity, the evidence presented here shows why such a marked tightening effect on the grain is secured. After adequate penetration of tan has been obtained, as indicated by a good shrinkage temperature in process and satisfactory fixation of ZrO_2 on analysis of the center area, the residual zirconium sulfate apparently is deposited on or near the surface as its basicity is increased in the neutralization step. A mechanism of this sort was suggested by Ranganathan and Reed (3) based on their experiments using hide powder, and the present results on unsplit heavy hide demonstrate very clearly how this effect can be used to advantage in practical tanning. Further work is planned to extend this approach to other leathers, and especially to study the distribution of zirconium when a small percentage is used in the retannage of chrome leather.

The effectiveness of only 1% citric acid in preventing excessive surface deposition is remarkable. There have been several recent references in the literature to the use of hydroxy acids in conjunction with zirconium tannage, but in most cases much higher amounts are described, going up from $\frac{1}{2}$ to 2 or even 3 moles per mole of ZrO_2 (3–6). With higher amounts of hydroxy acid, however, the tanning properties are lessened, and the cost of the process is increased.

The syntan retannage in all these experiments did not have any outstanding effect on the fixation or distribution of the mineral components of the tannage. However, in all cases the leather character was improved to a significant degree, particularly as regards greater mellowness.

SUMMARY

We have described how a problem in practical tanning utilizing insoluble sodium zirconium silicate led to a better understanding of the distribution of zirconium and silica in unsplit cowhide. The stratigraphic approach was

extended to compare a number of commercially available zirconium tanning salts with basic chrome sulfate. The use of only 1% citric acid at the start showed a remarkable effect in preventing excessive surface deposition of zirconium and silica. The new basic zirconium sulfate containing silica was found to be a good replacement for the method of acidification of sodium zirconium silicate, and also for the more expensive sodium zirconium sulfate.

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DISCUSSION

DR. ELL DEE COMPTON (Eagle-Ottawa Leather Co.): We are certainly indebted to Dr. Somerville for adding another block to the structure of the literature of zirconium tannage.

I am interested in Fig. 7, which shows a reduction in the zirconium deposit in the grain as the amount of citrate is increased. You made a comment, Dr. Somerville, that the use of the citrate gave a softer leather, and that even though the oil was reduced, it was softer. I wonder if there is an analogy here. I don't believe you used the word "masking". But is a citrate a good masking salt for zirconium and somewhat comparable to formate for chrome?

DR. SOMERVILLE: With reference to Fig. 7, the answer is that definitely it does have a masking effect. It actually holds off in the center, too. But I would definitely say that you have some masking effect—it does affect the distribution.

DR. COMPTON: Did you investigate masking salts other than citrate?

DR. SOMERVILLE: No. But we have, in the laboratory. Some are too severe. If you use too much masking agent, you convert your zirconium over into a nonionic form. But if it is used properly, it is a valuable tool in making the leather you wish.

DR. COMPTON: In your discussion you drew an analogy between zirconium leather and bark-tanned leather, saying that the zirconium leather is closer to bark leather than zirconium is to chrome. Of course the work at Leeds is along the same line.

We can add another bit of data, in the sense that some of the electrical properties of zirconium leather are closer to bark leather than chrome, and some of the rates of moisture pick-up and loss are also closer to bark leather than to chrome. Do you believe this is related to particle size or to weight, in any way? You brought in the degree of tannage idea. Perhaps that is it.

DR. SOMERVILLE: We do not have any figures on particle size or molecular weight of these different zirconium compounds. I would think that the distribution effects we have shown today are more connected with the deposition on the surface of the zirconium which has not been picked up in the primary tannage—the primary step of penetration—rather than on simply larger molecular size.

DR. COMPTON: Have you done any work adding the citrate with the zirconium, or has it always preceded it?

DR. SOMERVILLE: It is added at essentially the same time as the zirconium. It is 15 or 20 minutes before, but they are essentially at the same time.

DR. THOMAS C. THORSTENSEN (Lowell, Mass.): I would like to make one comment on the question of the masking effects with the zirconium. A few years ago I was working on a project on zirconium tannage with Dr. Theis, and we studied extensively the masking effects of various materials with zirconium. Included among them were the hydroxy acids—glycolic, citrate, and tartaric. As Dr. Somerville has pointed out, these are masking effects very much parallel to those found in chrome tannages. In the case of zirconium, the hydroxy acids are most effective and useful in this application.

ERNEST H. STEIN (Whitehall Leather Co.): Do the citrates have any beneficial effects on zirconium retanning of chrome, too?

DR. SOMERVILLE: We do not have any figures in that connection, but I am sure they do. If you are thinking of retanning chrome leather with zirconium, you are interested in the distribution. And I would think that if you add citric acid in that connection—and we have had some evidence—you will definitely get a more open surface. At least you tend to get a more mellow leather, because the surface is not so filled up. The subsequent fatliquor will penetrate better, and you can get on the whole a mellower leather using this masking effect in the case of a chrome retan.

STRENGTH INCREASES BY TENSION TANNAGES

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ABSTRACT

It is well known that tensile strength measurements, calculated for equal cross sections, give much higher results for individual collagen fibers than for mammalian hide in bulk, whether or not both are tanned or both are untanned. Also, it has been shown that when hide is forcibly pulled in one direction, a realignment of the fibers occurs, and this results in an increase in the tensile strength of the hide in the direction of the realignment.

A new and considerably improved technique of increasing tensile strength by mounting and tanning hide pieces under tension has been investigated, using bated or pickled cowhide and pickled sheepskin and various methods of tanning. Most of the pieces were chrome-tanned in a two-step process in which the chrome concentration was varied. Tension was applied during the first or second step or during both. An aldehyde-lignosulfonate combination tannage with stretching was also carried out. Characteristics of the stretched skins were compared with those of adjacent hide pieces that had been tanned in the same vessel but in a relaxed state. All the tannages under tension produced an increase in the parallel-direction tension strength (up to 12,000 lb in^{-2} or more) but at the expense of elongation, which was lowered by as much as 80%. It is believed that tension-tanned leather has potential military and civilian application.

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INTRODUCTION

Single collagen fibers and fiber bundles have been shown to be much stronger than hide or leather when the two are compared on the basis of equal cross-sectional area. Only a few data on the tensile strength of single fibers have been reported, namely:

Raw steerhide fibers (from forehead)	(in lb in^{-2})	70,000	(1)
Vegetable-tanned leather (from forehead)	"	36,000–41,000	(1)
Vegetable leather	"	14,000–21,000	(2)
Kangaroo tendon	"	47,000–58,000	(3)

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Most workers (4–8) have found it more practical to determine *mean breaking length* because accurate measurements of fiber cross sections are difficult to obtain. As far as untanned dry fibers are concerned, both types of measurement are in close agreement. The results from tanned fibers, on the other hand, vary widely, with the lowest figures (8.8–14.8 km.) encountered after tannage with formaldehyde (4,5), benzoquinone at pH 9.1 (4), and unmasked chrome liquor (5). However, there is no difference in results when small pieces of hide are first tanned and then teased (gross-piece technique) or when the fibers are teased from commercially tanned leather (6, 7).

It is obvious that the higher strength of fiber bundles is due to their parallel alignment. Therefore, a similar alignment of the fibers in leather should result in a substantial strength increase. Mitton and Nattrass (9) have shown this to be the case. By applying tension to pieces of hide while tanning them with chrome, they obtained strength increases in the order of 50% more than with hides normally tanned. As would be expected, these increases were observed only when the force was applied in the same direction as the tension during tannage. When the force was applied at right angles, there was a 12% loss in strength, on the average. This held true both for chrome- and vegetable-tanned leather, with the vegetable leather showing the greatest strength increases (up to 100%).

Mitton and Nattrass point out that tannage under tension "clearly offers substantial advantages for the production of leather whose strength must be high, particularly if the strength is required for pulls in one direction only." They cite leather belts as an example. We could also mention laces, gussets, and rucksack and ski bindings. However, they are aware that more data must be provided before the optimum conditions for tension-tanning can be decided upon and operations on a commercial scale undertaken.

Two Russian workers (10) concerned with protein fibers in general rather than with leather in bulk have conducted a related investigation that at present is accessible to us only as a brief abstract. Chemical Abstracts summarizes the results as follows: "Depending on the amount of stretching applied to the tannery process it is possible not only to fix a structure of the raw fibers but to obtain additional orientation of the molecular chains with a corresponding increase in the tensile strength of the fibers." Positive evidence of such realignment of molecular chains by mechanical forces was uncovered for an amorphous protein many years ago when Bjorksten and Champion (11) found that the rhythmical stretching and relaxation of air-dried gelatin sheets raises their tensile strength in the direction of stretch when they are acetaldehyde-tanned but not when they are untanned. The strength increase they obtained is modest when compared with that obtained by Mitton and Nattrass. Yet, by improving the earlier improvised stretching techniques, it has been possible to obtain strength increases of a much greater magnitude. The experiments demonstrating this are the subject of this paper.

EXPERIMENTAL

Materials.—Adjacent pieces of bated or pickled cowhide and of pickled lambskin were cut into suitably sized pairs with one of each pair being used as a control. The bated hide was preserved with Merthiolate and was pickled in the usual way before tanning. All hide material was wrapped in a polyethylene bag and kept under refrigeration.

Equipment.—A brass stretching frame (Fig. 1) consisted of two cylindrical bars, *A* and *B*, each 5.5″ long and 0.75″ in diameter, with one third of

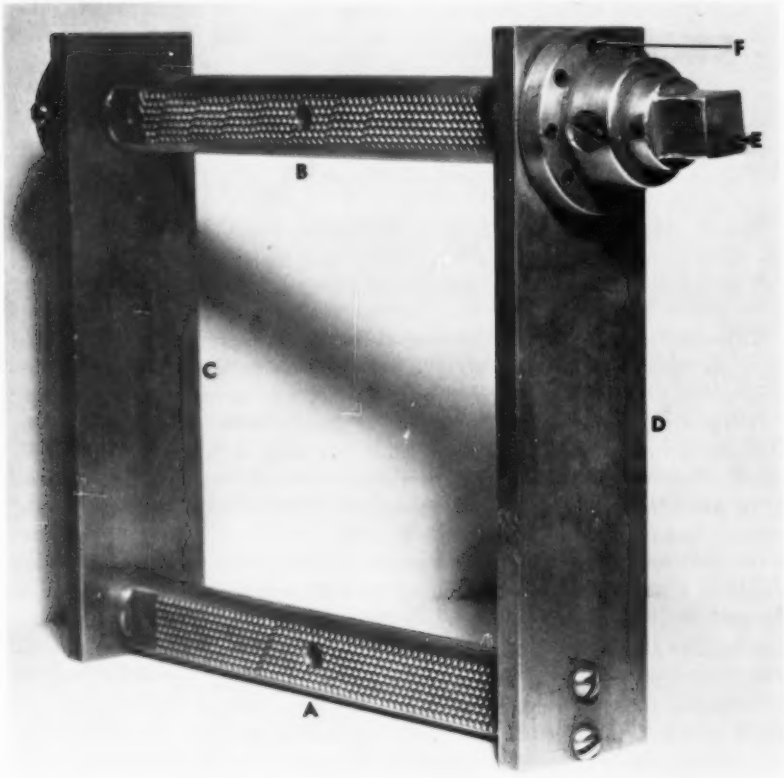

FIGURE 1.—Stretching frame.

A—Stationary bar
B—Rotating bar
C and D—Supporting plates
E—Turning knob
F—Hole for locking pin (Plate *D* has corresponding holes)

their surface flattened and knurled. Bar *A* was bolted securely between two plates, *C* and *D*. In a position 5″ from Bar *A* and parallel to it, Bar *B* was fastened between the plates but in such a way that it could be rotated by means of a $1\frac{1}{2}$″ extension (*E*) beyond the plate. A series of small holes in Plate *D* and corresponding holes penetrating a $\frac{1}{4}$″ disc (*F*) attached to Bar *B* outside the plate, permitted a pin to be inserted through both disc and plate in order to lock the turning crossbar at regular intervals.

The hide pieces, cut so that tension would always be applied parallel to the backbone, were attached first to Bar *A*, being held against the knurled surface by means of a brass plate (not shown) and machine screws that passed through threaded holes in the plates and punched holes in the hide. The other end of the hide was attached in a similar fashion to Bar *B*. A hand wrench applied to the protruding end (*E*) of Bar *B* turned the bar and thus stretched the hide. By marking the holes that the pin occupied at the point of the greatest possible tension, one could apply the same amount of tension to other pieces. The force required to turn the bar was measured by a torquemeter and was found to be 600 inch-pounds. Under this force, the hides were elongated about 25%.

In many experiments the size of the hide pieces was doubled by first fastening them to Bar *B* of the stretching frame, then looping them around Bar *A* and returning them to Bar *B*, which in this way secured both ends.

Tanning procedures.—A series of chrome tannages were carried out using an aged stock solution of 33% basic chrome sulfate (Tanolin R) containing 100 g/l of Cr_2O_3. Each chrome tannage was carried out in two steps.

In the first stage no masking agent or tension was employed. All the hide pieces were tanned in revolving glass jars using 0.5% or 1.0% of Cr_2O_3, based on their pickled weight. There was one exception, Tannage Method A, in which tension was applied during this "pretannage" (but not to the control) and for which 3.0% Cr_2O_3 was used.

For the second tannage the hide pieces, under stretch on the frame, were placed in a laboratory rocker of 3-liter capacity and moved up and down at the rate of 28 times per minute. The control piece in a relaxed condition was hung in the rocker next to the corresponding stretched piece. Immediately before use, the tan liquors were masked by adding 3 moles of sodium formate to 1 mole of Cr_2O_3, at room temperature. Except for Method A, in which only 1.5% of the pickled weight Cr_2O_3 was used, the quantities were 4.5% or 3% Cr_2O_3. The prepared solutions were sufficiently diluted to cover the stretching frame in the rocker.

During both tanning procedures the pH was slowly raised by adding small amounts of $NaHCO_3$ until a pH of 3.3 was attained for the *pretannage*, which took 24 hr. and brought the shrinking temperature (Ts) to 76°–78°C., or a pH of about 4.5 for the *second tannage*, which took about 48 hr. and brought

the Ts of the stretched pieces to about 100°–102°C. and of the controls to 103°–108°C.

After tanning, all specimens were washed for 10 min. in 25°C. running water and then air-dried.

While the same general procedure was followed for each tanning method, there were some exceptions, especially in fatliquoring. In all but Method A (in which there was no fatliquoring) the chrome-tanned pieces were fatliquored with 4% (of the pickled weight) of a mixture of equal parts of a cationic fatliquor and neatsfoot oil. The fatliquor was applied between the two tanning steps except in Method B, where it was applied after the retanning but before the air-drying.

An aldehyde and vegetable extract combination tannage, Method F, was also carried out, using a process similar to one recently developed for heavy leather by the Quartermaster Research and Engineering Laboratories (12). In this process one piece remained in the frame and the corresponding control piece hung freely in the tanning bath for 6 days. Equilibration with a solution containing Na_2CO_3 and $NaHCO_3$ in the ratio of 10 to 4 required 16 hr.; the aldehyde tannage using 3% technical formaldehyde (of the bated weight) required another 24 hr.; and the lowering of the pH to approximately 4.0 took 20 hr. more. A fresh liquor containing 20 parts liquid quebracho extract, 10 parts dry lignosulfonate extract, and 1.5 parts of a naphthalene sulfonic acid formaldehyde condensate (Leukanol ND) was used for the second tannage. This liquor was adjusted to 41° Bark. before being fed to the rocker, and was strengthened to 60° Bark. after 1 day, and to 80° Bark. the second day. For a period of 8 hr. out of the 24, the liquor was heated to 50°C.; thereafter it was allowed to resume room temperature. On the third day the pieces were rinsed and slightly fatliquored in a tumbling jar by the addition of 0.5% (of the bated weight) each of mineral oil, neatsfoot oil, and sulfonated cod oil.

Pieces of lambskin were also tanned under tension, using a single bath of formate-masked basic chromic sulfate and raising the pH to 4.5 with $NaHCO_3$. The liquor was rocked for 32 hr. and then left standing for 16 hr. before relaxation of the tension and fatliquoring in a revolving jar.

In addition, pieces of pickled cowhide and lambskin were fastened to a rectangular-type stretching frame that permitted them to be stretched in two directions (parallel and perpendicular to the backbone) as in a fabric stretcher. While subjected to such stretching, the pieces were tanned by the same method as C, D, and E.

These tanning procedures differed from those of Mitton and Nattrass in (*a*) employing cow or sheep skin rather than calf, (*b*) reducing the "exceptionally high" ratio of tanning bath to hide to about 10:1, (*c*) being more rapid—3 days instead of 8 for the chrome, and 6 instead of 28 for the vegetable

tannage—and (*d*) producing chrome leather with a Ts of 100°–102°C. instead of 91°–93°C. A similar comparison cannot be made for the vegetable-tanned leather because of lack of data from Mitton and Nattrass.

Testing methods.—All pieces were air-dried and conditioned to 50% relative humidity and 73°F. before being tested for strength. Breaking load, tensile strength, elongation, and stitch tearing strength (when size of piece permitted including this test) were obtained in accordance with Federal Specification KK-L-311, except that the dumbbell specimen was only 4" long and 0.25" wide at the "waist". Bursting load was measured by means of a modified Scott Tester, using a 1/8" plunger and a 3/8" orifice. All strength measurements were taken in the direction of stretch. Ts measurements were obtained with a Transverse Shrinkmeter (13).

TEST RESULTS

Cattlehide leather

Tensile strength.—Physical data for the cowhides tanned under tension are given in Table I. Data for the unstretched control specimens are not given in order to keep the number of columns to the minimum, but they were used in computing the percentages of increase or loss. Strength measurements are given both in pounds and in pounds per square inch. This is because stretching causes loss of thickness to varying degrees. On an equal cross-sectional basis, the chrome tannages A and B, the latter conducted on flanky belly pieces, and the aldehyde-vegetable tannage F gave especially large strength increases.

Table I shows that the average strength of tension-tanned leather depends to some extent on thickness. Very thick pieces such as were used in Method D, which was carried out on particularly heavy shoulder pieces, may exceed breaking loads of 300 lb., or tensile strengths of 10,000 lb in^2. Light pieces, on the other hand, such as were used for Methods C and E, may reach a breaking load of only 100 lb., or a tensile strength of only 6,000 lb in^2; these pieces were thinner than the rest because they had been stretched after removal of a flesh split.

In Methods D and E, half of the control pieces, after having been tanned in the relaxed state, were mounted on the stretcher before washing and kept there for the same length of time (48 hr.) as the pieces that had been tanned under tension. As Table I shows, this "post-stretching," identified as Methods D_2 and E_2, is not as effective as the tension tannage.

Individual test results not shown in the table revealed that the greatest increases in strength were obtained from that part of the hide at the outer edge of the stretched part. There the fibers were subjected to a greater pull than in the center, and as a visible indication of this extra pull, all specimens

TABLE I

CHARACTERISTICS OF CATTLEHIDE LEATHER AFTER TANNING UNDER TENSION

(Force applied in same direction as tension)

Tannage Method	Chromic Oxide (% of pickled weight) First Stage	Chromic Oxide (% of pickled weight) Second Stage	Fatliquor Applied	Thickness	Breaking Load	Tensile Strength	Elongation at Break	Increase: Breaking Load	Increase: Tensile Strength	Loss: Elongation	Loss: Thickness	Av. No. Measurements*
				oz.	lb.	lb/in2	%	%	%	%	%	
A	3†	1.5†	None	6.7	325	12,200	—	207	294	—	21	3
B	1	3†	After retan	4.8	158	8,090	28	110	236	64	34	7
C	1	3†	Between pretan and retan	3.3	85	6,470	18	157	177	80	14	20
D_1	0.5	4.5†	" " "	7.6	303	10,100	29	106	148	76	22	7
D_2	0.5	4.5	" " "	8.8	284	8,610	32	93	111	74	10	7
E_1	0.5	4.5†	" " "	4.4	100	5,760	18	127	135	67	4	7
E_2	0.5	4.5	" " "	4.2	56	3,450	20	27	41	64	9	7
F	Aldehyde-extract†		After retan	8.9	300	8,020	24	124	210	nil	31	7

*Except for thickness where from 2 to 3 times as many measurements were made.
†Signifies tanning stage or stages where tension was applied.

assumed a slightly constricted form during tannage. Evidently, the fibers in that area had become not only better oriented but subject to greater extension. For instance, the peak tensile strength from Method B was 11,260 lb/in² at the outer edges. Two specimens from the same location exhibited tensile strength values of 10,070 and 6,950 lb/in² and breaking loads of 365 and 128 lb. respectively after Tannage Methods D_1 and E_1. This "edge" effect was evidently missing from the British experiments, which were conducted on pieces over 50% wider than ours, and its absence explains—at least partially—why the strength increases observed were more modest than those found in this study.

Mitton and Nattrass observed that breaking load and tensile strength taken at right angles to the direction of the applied tension are both considerably decreased in comparison with the controls. This observation was confirmed by a few tests not included in Table I.

Elongation.—The tension-tanned chrome leather pieces of Methods A to E broke at elongations varying from 18% to 32%, or at values from 64% to 80% lower than the elongations at break of the controls. The degree of elongation observed apparently is unrelated to the tensile strength. In Method F, the elongation at break was the same both for the tension-tanned and control pieces, a result that reflects the well known rigidity of leathers tanned with vegetable extracts.

TABLE II

BURSTING STRENGTH OF TENSION-TANNED AND CONTROL PIECES OF CATTLEHIDE LEATHER

Tannage Method	Bursting Strength		Degrees of Freedom	*F* Value
	Tension-tanned lb.	Control lb.		
A	190	184	1,7	0.20
B	125	119	1,4	0.36
C	43	44	1,16	0.62
D_1	112	125	1,6	1.7
E_1	90	73	1,14	9.9*
F	245	240	1,26	0.12

*Significant at the 99% level ($F = 8.91$); other F values not significant.

Bursting strength.—The results of the bursting strength tests are presented in Table II. An analysis of variance was performed on each of the tannages. As the table shows, the F values received are very low and not significant except in the case of Tanning Method E_1, carried out on grain splits. The bursting strength of the tension-tanned piece was more than 20% greater

than that of the control. A second tannage of grain splits (C) showed no appreciable difference in strength between the tension-tanned and the unstretched piece. This inconsistency may have been caused by the degree of splitting or by positional differences.

Stitch tearing strength.—The results of the stitch tearing test (given to only three tension-tanned cowhide pieces and their controls) are given in Table III. The tension-tanned pieces were found to be considerably weaker than

TABLE III

STITCH TEARING STRENGTH OF TENSION-TANNED AND CONTROL PIECES OF CATTLEHIDE LEATHER

Tannage Method	Direction of Test	Stitch Tearing Strength		Difference X	Deviation x
		Tension-tanned X_1 lb.	Control X_2 lb.		
A	Parallel	61	90	29	1
	Perpendicular	62	106	44	16
D_1	Parallel	80	154	74	46
	Perpendicular	100	126	26	− 2
E_1	Parallel	62	45	−17	−45
	Perpendicular	46	58	12	−16
Total		411	579	168	0
Mean		68.5	96.5	28	

By statistical analysis, on a paired comparison basis (see G. W. Snedecor, *Statistical Analysis* [Iowa State College Press, 1948] page 44), a t value of 2.24 is obtained, which is significant at the 95% level.

the controls, with the exception of the grain splits tanned by Method E_1 and tested in the parallel direction. The number of measurements was too small for a statistical analysis of directional effect; however, a paired comparison between tension-tanned and control pieces can be made, and such a comparison indicates a very significant tendency toward loss in stitch tearing strength by tanning under tension. This was not unexpected in the light of previous work on the various types of tears (14, 15).

Neither stitch tearing properties nor bursting strength was studied by Mitton and Nattrass.

Chemical properties.—For the tension-tanned chrome leathers a minimum Ts of 100°C. was considered adequate. In order to obtain this Ts the control pieces tanned in the same liquor generally had a 2°–3°C. higher Ts. Oc-

casionally, however, differences as much as 8°C. were observed between the tension-tanned and control pieces. For the aldehyde-vegetable tension-tannage the difference was 14°C., and for that reason this leather was fully analyzed, with results given in Table IV.

TABLE IV

CHEMICAL COMPOSITION OF ALDEHYDE-VEGETABLE TENSION-TANNED LEATHER

	Tension-Tanned	Control
Water solubles, %	6.4	12.1
Hide substance, %	70.0	55.3
Grease, %	1.7	2.5
Water-insoluble ash, %	0.3	0.3
Degree of tannage	31	54
Acidity (pH)	4.8	5.0

The difference between the stretched and unstretched pieces is shown to be considerable. The stretched showed fewer water solubles and less grease, which would be expected from the pulling of the fibers closer together, resulting in a markedly lower degree of tannage. These differences might have been minimized by extending the duration of the tannage and by using a more economical liquor-hide ratio.

New Zealand lamb.—The test results from the pieces of lambskin tanned under tension are shown in Table V. The breaking load and tensile strength were again found to be many times greater for the tension-tanned leather than for the controls. The differences are of the same order of magnitude as those obtained with cowhides of similar thickness, although single measurements of breaking load exceeded 140 lb. and 10,000 lb in^2 of tensile strength.

Cowhide and lambskin subjected to two-way stretch.—Parallel and perpendicular (relative to the backbone) breaking loads of air-dried pieces of pickled cowhide and lambskin that had been stretched in two directions while being tanned according to Methods C, D, and E were compared with those of the control pieces, with the results (averages of 6 measurements per value) shown in Table VI.

TABLE V

CHARACTERISTICS OF CHROME-TANNED SHEEPSKINS AFTER TANNING UNDER TENSION

(Force applied in same direction as tension)

	Thickness oz.	Breaking Load lb.	Tensile Strength lb/in²	Bursting Strength lb.	Elongation %	Increase		Loss Elongation %	Average Number Measurements
						Breaking Load %	Tensile Strength %		
Tension-tanned	3.3	94	7,260	39	13	261	245	72	7
Control	3.1	26	2,110	30	47				7

TABLE VI

BREAKING LOADS

	Cowhide lb.	Lambskin lb.
Parallel direction		
Tension-tanned	13	15
Control	16	24
Perpendicular direction		
Tension-tanned	12	12
Control	17	19

These figures show that, without exception, two-way stretch failed to orient the fibers for increased strength and actually caused strength losses to varying degrees.

CONCLUSIONS AND RECOMMENDATIONS

The results of tanning under one-way tension clearly indicate highly significant strength increases in the direction of the applied tension. In unsplit chrome- or vegetable-tanned leather, tensile strength values of approximately 10,000 lb in^2 are feasible. Cowhide split to 3 or 4 oz., and also pickled lambskin, can reach average tensile strength values of about 7,000 lb in^2 and average breaking loads of about 100 lb. Tanning under tension, therefore, distinctly appears to be a possibility for upgrading inferior stock or for producing leather of exceptional strength.

The small-scale experiments reported in this paper were undertaken primarily to create new interest in tension tannage. The next step would be the construction of increasingly larger stretching frames in which hide pieces could be tanned under tension. It is believed that the building of a practical stretch-tanning unit would not be unsurmountable. The continuation of work of this kind in the Quartermaster laboratories is impossible, however, because of the curtailment of funds.

The future will determine whether the stretching technique can also be used in studies of a theoretical nature, especially in studies of those phenomena that depend mainly on weave and cohesion of fibers rather than on spatial changes in the protofibrils or molecular chains.

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Tribute was paid, in the December, 1935, issue of *JALCA* to Fraser Muir Moffat (1868–1935) who passed away suddenly on November 7. "All things considered," said the *Journal*, "he had accomplished more for the immediate and future good of the American Leather Industry than has any man in its long history." In 1921 Mr. Moffat became President of the Tanners' Council and to devote his entire time to that work he closed up his business. A listing of his accomplishments and achievements during his Tanners' Council work is reported by George D. McLaughlin.

Published in *JALCA*, December, 1935, were the following papers:

The Action of Microorganisms on Vegetable Tanning Materials, II. A General Consideration of the Factors Influencing Biological Activity, by William P. Doelger, with the technical assistance of Robert Partridge and Lester Schreiberg, Dept. of Leather Research, Foundation Tanners' Council of America, Institute of Scientific Research, University of Cincinnati; Studies in Chrome Liquors, Part V. The Effect of Various Factors in Manufacture of Sucrose-reduced Liquors upon the Cr_2O_3 and Oil Take-up of Pickled Hide Powder, by Edwin R. Theis and E. J. Serfass, from the Dept. of Chemical Engineering, Lehigh University, Bethlehem, Pa.; Deterioration of Vegetable-tanned Leathers Containing Sulphuric Acid and Glucose, by Everett L. Wallace and Joseph R. Kanagy; and Effect of Acid on Leather—A Summary, by Warren E. Emley.

LIFE LINES

Brief Biographies of Our Contributors

HUBERTINA D. CLAYTON was graduated from the University of Pennsylvania in Philadelphia with a master of science degree in biochemistry after having completed her undergraduate work in biology and chemistry at Seton Hill College, Greensburg, Pa. She has been active in research on tanning and leather development since 1952 in the Leather Branch of the Quartermaster Research and Engineering Command.

MISS H. R. FRANECKE has been employed for sixteen years in the analytical section of the Leather Chemicals Laboratory of the Rohm & Haas Company, specializing in the analyses of natural and synthetic tanning materials and a wide variety of leathers.

L. V. HETZEL has been in charge of the Experimental Tannery at the Bridesburg Laboratories of the Rohm & Haas Company for ten years. He is responsible for application tests on new products for beamhouse and tanning, taking these from a laboratory scale to the stage where they are ready for tannery trials.

CHARLES W. MANN, a frequent contributor to this *Journal*, is a graduate of George Washington University. In 1941 he began working for the U. S. Department of Agriculture on hides, tanning materials, and leather. Five years later he joined the staff of the Bureau of Standards where he worked for two years on the properties of leather, with emphasis on military leathers. In 1948 he joined the Quartermaster Research and Development Division as head of the Leather Section. Until recently he has been chief of the Leather, Footwear and Handwear Branch of the Textile, Clothing & Footwear Division, Quartermaster Research and Development Command. Now he is on the staff of the Lowell Technological Institute Research Foundation.

DR. LUDWIG SELIGSBERGER, born and educated in Germany, worked both there and in Turkey before coming to the United States in 1937. After many years as chief chemist of the New Castle Specialty Division of Allied Kid Company in Wilmington, Delaware, he joined the Leather Technology Laboratory Branch of the Quartermaster Research and Engineering Center, Natick, Mass., where he is now Section Chief in the Leather and Footwear Branch of the Textile, Clothing and Footwear Division. He has published several papers in this *Journal*.

IAN C. SOMERVILLE obtained his B.Sc. and Ph.D. degrees at the University of Edinburgh, Scotland. He has been with the Rohm & Haas Company for thirty years and is head of their Research and Development Laboratory on Leather Chemicals. His contributions to *JALCA* include papers on sulfiting of quebracho, on syntans, and on zirconium tannage. He joined ALCA in 1935 and received the Alsop Award in 1950.

JESSE WENDKOS has been associated with the Rohm & Haas Company for thirty years, most of the time in charge of the analytical section of the Leather Chemicals Laboratory. He has been coauthor with Dr. Somerville of several papers on the analysis of zirconium tanning materials and leathers and has served on the Mineral Tannage Committee of our Association for eight years.

. . . At the International Conference on Scientific Information (Washington, 1958) . . . past studies of the informational needs and habits of scientists were reviewed, and new types of studies were presented and discussed. One of the few points on which consensus was reached was that there was increasing dependence on channels other than the traditional scientific journal-abstracting publication-indexing service sequence, that has been termed here, the formal channel. The reasons for this change could not be definitely stated, as sufficient information was not available; but it was agreed that a number of factors play a role.

"HALF-LIFE" OF INFORMATION

The more active the field of research, the shorter the useful "half-life" of the average new item of information. The half-life of information is a concept borrowed from radioisotope terminology and is that period in which will be made one-half of all the uses of a given piece of information. This half-life varies widely from one branch of science or field of research to another but, in general, is longer for the biologic than for the physical sciences. In certain special new fields of physics, it is said to be only a matter of months. In the more dynamic areas of medical research, for example, new drug development, most of the value of a new finding may have been extracted in only a year or two.

PUBLICATION SLOWS PROCESS

Since publication by traditional means usually requires at least six-18 months after completion of a research project, it is obvious why researchers make every effort to anticipate the formal publication process and to obtain information through other channels while it is still of maximal value in their own work. This is undoubtedly the chief reason why scientific meetings are assuming greater importance and why the practice of exchanging information in oral and written form before formal publication seems to be increasing.

—R. H. Orr, M.D. "Communication 'Unpublished' Information." *Research/Development Magazine*, September, 1960.

ALCA NEWS

Proceedings, Reports, Notices, and News

of the

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The Officers and Council of the Association join the editorial staff in extending to all members the warmest of season's greetings and sincere wishes for a happy, prosperous and peaceful New Year.

Obituary

EDWIN E. OCHS

Edwin E. Ochs was born November 5, 1901, in New York City. After graduation from high school he joined the Wallerstein Laboratories in New York City. During his employment there he continued his education by attending the Brooklyn Polytechnic Institute. As an assistant in the research laboratories at Wallerstein he acquired a thorough knowledge in the field of enzymes and their practical industrial applications. He became especially interested in the study of proteolytic enzymes and their use in the leather field for unhairing and bating. During these investigations he became very well acquainted not only with beamhouse and bating procedures but also with subsequent stages of leather manufacturing.

His great interest in leather-making led him to accept a position as chemist with John R. Evans and Company in Camden, N. J., in October, 1934. He continued his association with this company until his sudden death on July 4, 1960. During this period he organized their production control and research laboratories. He also trained his assistants so thoroughly that they advanced to responsible positions in the organization.

His research work at Evans concerned all steps in the manufacture of leather. In 1937 he developed a process for treating hides and pelts comprising the treatment of limed skins and hides with prepared enzymatic bating materials and carbon dioxide which was patented February 16, 1939 (U. S. patent 2,147,542). He also recorded on May 29, 1946, an improved process for preserving hides and skins. At the Golden Jubilee Meeting of our organization in Cincinnati on June 8, 1953, he presented a paper "Modern Practice in Two-Bath Chrome Tanning." "Eddy," as he was known to all our members, joined ALCA in 1937 and was active in the affairs of our association. He also had many other interests. He lived in Riverton, N. J., where he was a vestryman in Christ's Episcopal Church and treasurer of the Sunday School. He also was a member of the Riverton Country Club.

We shall all miss Eddy with his sunny disposition and wish to extend our sincere sympathy in their bereavement to his widow and daughter.

Julius Pfannmuller

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ABSTRACTS

Zirconium-Vegetable Combination Tannage. T. S. Ranganathan and Y. Nayudamma. *J. Indian Leather Technol. Assoc.*, **8**, 125–29 (1960).—Pieces were tanned with Zr and retanned with wattle or myrobalans, or tanned with wattle or myrobalans and retanned with Zr salts. The abrasion resistance of these leathers, especially the Zr-retanned specimens, was appreciably higher than that of straight vegetable controls. Water solubles were lowered considerably, and shrink temperatures were raised. A method is described for producing sodium zirconium silicate from Indian zircon sand, and tanning by a procedure similar to that of Somerville and Rau (*JALCA*, **51**, 541–54 [1956]). H.B.M.

Studies on Post-Mortem Changes. I. Effect of Post-Mortem Changes on the Swelling of Hide and Leather. S. C. Nandy and S. N. Sen. *Bull. Central Leather Research Inst. Madras.*, **6,** 478–83 (1960).—Freshly slaughtered cowhide pieces were washed, fleshed, shaved to remove hair, and stored in closed containers for 2 to 72 hr. at 30°C., then swollen in saturated lime solution. Swelling declined to a minimum for a 5-hr. staling period (cf. McLaughlin, *JALCA*, **16,** 435 [1921]) but increased with longer staling periods and was greater than for freshly flayed hide when the staling period exceeded 24 hr. Tests were also made on specimens that were dried out after staling for 2 to 48 hr. and on Cr leather made from specimens staled for 3 to 72 hr. These swelling tests were made in veronal buffers at pH values from 2.5 to 8.45. The dried hide pieces showed increased swelling with duration of staling at all pH values. The Cr leathers showed little difference in swelling for staling periods up to 48 hr., but a marked increase in swelling for 72 hr. staling. These results probably reflect a general loosening of the fiber structure caused by bacterial attack, allowing the skin to imbibe more water. H.B.M.

The Investigation and Evaluation of Fatliquor Products and Leather Auxiliaries. F. Stather and G. Weigand. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14,** 103–13 (1959).—The Panzer-Niebuer separation (*Leder.* **3,** 219 [1952]) of fatliquors into the emulsifier (soluble in dilute alcohol) and the emulsified constituent (soluble in petroleum ether) has been studied extensively and found to have wider applicability than generally supposed. Application of this separation to known mixtures of mineral oil and fish oil, emulsified with soap or Mersolate, gave results within 1 or 2% of the correct values. The separated emulsifier can be analyzed for combined SO_3, or for N in the case of a cationic emulsifier, and the results are said to mean more than the same determinations made on the whole fatliquor. Likewise the separated emulsified component can be analyzed like any fat, particularly for unsaponifiable matter, free fatty acid, etc. Data are given for 9 commercial fatliquors analyzed by the Panzer-Niebuer separation and by the usual methods for sulfated oils. The values for total fat, obtained directly by the former method, are considered more reliable than those obtained by difference. The separation was carried out at pH values from 3.0 to 9.4 on a variety of fatliquors. For those with anionic emulsifier, the quantity of emulsifier found declined with pH value, but the reverse was true for cationic emulsifiers. The separation was applied to 5 leather auxiliaries (soaking assists, degreasing materials, etc.), which differed from fatliquors in containing mostly emulsifier and water, with very little emulsified fat. If the usual solvent pair (petroleum ether and 70% alcohol) does not give a good separation for a specific material, more dilute alcohol may be employed, or the petroleum ether may be replaced with benzene. H.B.M.

The Black Dyeing of Persian Lamb Skins. F. Stather, H. Herfeld, S. Walther, and U. Stather. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14,** 115–29 (1959).—The commercial process is to soak the skins in an incomprehensible decoction containing logwood, sumac, galls, iron filings, verdigris, $CuSO_4$, $FeSO_4$, and salt, after which the skins are hung up to develop the color by air oxidation. By examining the effect of each factor a simplified procedure was worked out, eliminating the iron filings and verdigris. Sumac

and galls were found to be almost interchangeable, but ellagitannins could not be used. Copper alone gave the most intense black, but some $FeSO_4$ helps to throw the shade from brownish toward the blue. It is necessary to treat the skins with the liquor at 37°C. for 24 hr. to get uniform coloring to the base of the hair shafts. A pretreatment with dilute NaOH did no good. The skins must be freed from excess liquor before hanging and must be hung for 24 hr. in a moderately damp atmosphere to avoid drying out. Finally the skins are washed and neutralized. H.B.M.

The Chrome Dressing of Fur Skins with Formate-masked Chrome Liquors. H. Herfeld, S. Beyer, and R. Andree. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14,** 131–37 (1959); cf. Stather *et al.*, abstr., *JALCA*, **51,** 396 (1956).—Tests were carried out with lamb and rabbit skins. The subjective properties of the furs are improved by adding 2 to 3 mols HCOONa per mol Cr to the tanning bath. Best results were obtained using 1 to 1.5% Cr_2O_3 in a 50% basic liquor, with no neutralization during tanning. Pickling is unnecessary for lamb skins but is required for rabbit skins to open up the fiber structure. After pickling, rabbit skins should be neutralized with NH_3 to pH 4. The final shrink temperature should not exceed 80°–85°C., otherwise the product is too leathery. Analytical and physical test data are given. H.B.M.

Investigations on the Useability in Tanning of Chemically Debarked Spruce Bark. E. Zieger and O. Blossfeld. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14,** 139–43 (1959).—Spruce bark from trees that were girdled and treated with Na_3AsO_3 contained only about 1/3 as much tannin (4 months after treatment) as control trees, whether the tannin was extracted with water or with dilute Na_2SO_3. The tannin, what there was of it, produced satisfactory leather. The As content of the bark or extract did not constitute a serious health problem. Cf. Beebe *et al.*, *JALCA*, **51,** 266 (1956). H.B.M.

Improvement of the Sweat Resistance of Cattle and Pig Insole Leather. F. Stather, H. Herfeld, G. Reich, and H. Moser. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14,** 5–26 (1959).—The sweat resistance of commercial East German insole leathers was measured by the methods of Grassmann and Stadler (*Leder*, **4,** 213 [1953]) and Vago and Fekete (*Leder*, **8,** 137 [1957]); the latter is quicker but less sensitive to small differences. The leathers tested were generally unsatisfactory, especially when more than the compulsory 50% of syntan was used in the tanning blend. Pig and cattle hide leathers differed little in sweat resistance. Tests of experimentally tanned leathers showed that straight vegetable-tanned leathers—either spruce alone or chestnut-oak-quebracho blend—were better than leathers tanned with the commercial blend containing 50% syntan. The resistance to sweat of straight syntan leathers varied with the kind of syntan. Thirty experimental pig insole leathers were tanned by various modifications of the following procedures: straight Cr tannage; Cr pretannage and vegetable-syntan retannage; straight vegetable-syntan tannage; vegetable-syntan tannage retanned with Cr, Al, amphoteric tanning agents, or resin tanning agents; straight urea-HCHO tannage, and urea-HCHO tannage with Cr or vegetable retannage. Sweat resistance was assessed from degree of retention of initial area after 10 times wetting

with water or artificial sweat and drying. The shrinkage after treatment with artificial sweat increased with pH value and was accompanied by considerable detannization, 10 to 30% loss in tensile strength, hardening, loss of fat, and discoloration, but improvement in water absorption. The straight vegetable-syntan leather had the poorest sweat resistance, and the straight Cr and straight resin leathers the best, but these latter were unsuitable otherwise. Cr pretannage produced less improvement than retannage with Cr or Al salts; also Cr pretannage resulted in poorer yield figures. Retannage with amphoteric tanning agents or with resin tanning agents was generally ineffective, except retannage with urea and HCHO. All things considered, the Cr and Al retannages are preferred, but they have the drawback of producing inferior color. H.B.M.

Shortening the Duration of Sole Leather Tanning through Chrome Pretannage and Vegetable-Syntan Liquor Tannage at Elevated Temperature. F. Stather, G. Reich, J. Überla, and H. Moser. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14**, 53–59 (1959).—A detailed description of a semiworks scale experiment, in which a series of about 60 small lots, each of 50 kg. of cattlehide croupons, was pickled, lightly Cr-tanned, and then tanned in a set of 12 liquors for 50 days. Each lot received 30% pure tannin, consisting of 50% of a mixture of 3 syntans, and 50% of a mixture of 5 vegetable tannins, mostly spruce and oak wood. The tail liquor was discarded after every 3 lots. Initially the temperature was 30°C. in the first 4 liquors, 40° in liquors 5 and 6, and 45° in liquors 7 to 12, but this resulted in damage to the leather, perhaps because of the high proportion of syntan; consequently the temperature was dropped to about 30°C. throughout. The leather produced was acceptable, but degree of tannage and yield value fell off as the experiment progressed, supposedly because the amount of tannin given was too low.
H.B.M.

The Dyeing of Pig Suede Leather. F. Stather, H. Herfeld, H. Moser, and K. Härtewig. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14**, 61–76 (1959).—Procedures for dyeing pig suede leather, prepared as previously described (abstr. *JALCA*, **53**, 653 [1958]) were investigated. The skins were treated with ammonia, acid dye, HCOOH, and a light fatliquor in that order. Data are given on degree of exhaustion of the dye bath when using different amounts of NH_3 and HCOOH. Adding part of the acid between dye feeds and adding the fatliquor before acidification are disadvantageous. Treatment with a cationic auxiliary after dyeing gives better exhaustion but nonuniform color and decreased water repellance. Topping with basic dyes is unsatisfactory. The retannage with cationic Cr, after the leather has been dried, is again shown to be necessary in order to give the leather sufficient affinity toward anionic dyes, but this retannage can be replaced by treatment with a cationic synthetic resin. Basic Al compounds did not work as well. The dyeing temperature should not exceed 70°C. The suitability of 44 different dyes for coloring suede was investigated. H.B.M.

Imparting Luster to Pig Suede Leather. F. Stather, H. Herfeld, H. Moser, and K. Härtewig. *Ges. Abhandl. deut. Lederinst. Freiberg/Sa.*, **No. 14**, 77–86 (1959).—The purpose of this operation is to improve the depth, uniformity, and rub-fastness of the color, and if possible to improve the suedev

feel. This can be brought about by 4 different types of treatment: application of colorless materials that alter the optical properties of the surface, basic dye solutions, pigmented nitrocellulose finish, and pigmented casein finish. The desired effect can be attained by applying sulfated oil with a low "water penetration no.", a soap emulsion of linseed or olive oil, or aqueous solutions of gelatin or HCHO, by steaming, or by applying very small amounts of the nitrocellulose finish. The casein finish is less satisfactory, and application of basic dyes is disadvantageous. Highly sulfated oils render the leather too water-absorbent. H.B.M.

The Separation and Characterization of the α and β Components of Calfskin Collagen. K. Piez, E. Weiss, and M. Lewis. *J. Biol. Chem.*, **235**, 1987–91 (1960).—After purification of collagen-containing acetic acid extracts, parent gelatin was prepared by heating acidic solutions of the purified calfskin collagen to 40°C. Sedimentation in the ultracentrifuge at pH 4–8 of the collagen warmed to 40°C. for a few minutes produced two peaks. Sedimentation coefficients at infinite dilution were 3.245 and 4.415 for the α and β components, respectively. These sedimentation coefficients are consistent with previously suggested molecular weights of 80,000 (α component) and 160,000 (β component). Chromatography of the parent gelatin on carboxymethyl-cellulose columns (37°C.) separated the two protein components which after rechromatographing (estimated purity of fractions 90%) were examined for amino acid analysis. Amino acid-wise the α and β components are similar but not identical. The hydroxyproline and glycine content in the two components differed by 5% and 2% respectively, while other amino acids (aspartic acid, glutamic acid, proline, alanine, valine, isoleucine, leucine, hydroxylysine, lysine, and histidine) differed by larger amounts (from 8% to more than twice). J.M.C.

Condensed Tannins. IV. The Distribution and Deposition of Tannins in the Heartwoods of *Acacia Mollissima* and *Schinopsis Spp.* D. Roux and S. Evelyn. *Biochem. J.*, **76**, 17–23 (1960).—Procedure for removal of tannins from heartwood and subsequent paper chromatographic isolation of (+) catechin and gallic acid from heartwood extractives of *Schinopsis Spp.* is described. Vertical sampling of the heartwood of *S.-quebracho-colorado* shows a vertical cone effect present in the distribution of the average molecular weight of the tannins and concentration of nontannins in the upper portion of the tree. The heartwood tannins of *A. Mollisima* consist of a gradation over the molecular weight range 290–1650 in which the yield of anthocyanidins decreases with increasing average molecular weight. J.M.C.

Condensed Tannins. V. The Oxidative Condensation of (+) Catechin. S. Evelyn, E. Maihs, and D. Roux. *Biochem. J.*, **76**, 23–7 (1960).—The autooxidation of (+) catechin in aqueous solution is relatively slow and is accompanied by formation of tannins through condensation. The synthesis of tannins is also accompanied by degradation resulting in an over-all complex reaction. J.M.C.

Development in Combination Tannages. I. Factors Influencing the Strength of Wattle-retanned Chrome Leather. R. L. Sykes and D. A. Williams-Wynn. *J. Soc. Leather Trades Chemists*, **44**, 326–35 (1960).—The effects of a number of process variables on physical properties of wattle-retanned chrome leather made from medium weight, wet-salted cattlehides were studied. Buckle tear test and grain crack test (lastometer) were used for assessment of leather quality. The choice of processing conditions had considerable effect, the following ones tending to make for a stronger and more acceptable leather: (1) mellow liming with a minimum of mechanical action, (2) chrome tannage with about 1.25% 33 basic Cr_2O_3, (3) introduction of a chrome complexing agent into the leather during neutralization, (4) application of part of the fatliquor immediately after neutralization, (5) retanning in as short a time as possible, but preferably addition in two lots, and (6) adjustment of drying conditions and fatliquor composition so that staking can be minimized or eliminated. S.D.

The Rapid Tannage of Light Leathers. J. H. Atkinson and N. J. Cutting. *J. Soc. Leather Trades Chemists*, **44**, 318–25 (1960).—Degreased, pickled, brine-washed sheepskins were pretanned with 10% synthetic tannin (condensed cresol sulphonic acid type) by drumming for 30 minutes. The main tannage was then accomplished by rapid addition of 50% mimosa extract at 200° barkometer strength and drumming until penetration was complete (normally 2 to 2½ hours). The above procedure, suitably modified, can be applied to other classes of light skins and with other tanning extracts. Because the method involves drumming in a small amount of liquor, the flexing of the skins and the frictional heat developed are greater than in conventional tannage. A drum speed of 3–4 revolutions per minute is recommended for the normal-sized drum. Optimum operational conditions for this type of tannage are discussed. Promising results were also obtained with rapid tanning of limed pelt without previous deliming. S.D.

Reflection on the Straight-through Machine Idea. W. R. Gaythwaite. *J. Soc. Leather Trades Chemists*, **44**, 335–44 (1960).—The author attempts to assess the economic advantages and technical problems associated with application of the automation principle to the leather-making process. S.D.

Experiments on Wool from Copper-deficient Sheep. I. Some Physical Measurements on Intact Fibers and Experiments on the Dissolution and Fractionation of Oxidized Wool. R. W. Burley. *Textile Research J.*, **30**, 473–84 (1960).—Copper-deficient and normal wools were oxidized with H_2O_2 in formic acid, and the dissolution in aqueous buffer solutions of the oxidized wools was examined. Further evidence that the copper-deficient wool is deficient in a sulfur-rich fraction was obtained. This absence may be responsible for the fact that copper-deficient wool has a faster rate of extension in water under constant load than normal wool and also a greater diameter swelling in formic acid. **II. Amino-Acid Composition of Oxidized Normal and Copper-deficient Wool Fractions and Further Attempts at Fractionation.** R. W. Burley and F. W. Horden. *Textile Research J.*, **30**, 484–89 (1960).—The amino acid compositions of oxidized normal and copper-deficient wool and of two components separated from them at pH 6 were measured by

ion-exchange chromatography. The soluble and insoluble components had different amino acid compositions, the chief difference being in the amounts of cysteic acid, aspartic acid, proline, leucine, and lysine. The composition of the insoluble component was the same whether it was derived from normal or copper-deficient wool. The soluble component from normal wool had more cysteic acid than that from copper-deficient wool. Attempts at fractionating the "soluble" components gave indications of heterogeneity. C.L.D.

Method of Removing the Outer Layers of Animal Fibers. J. W. Snaith. *Textile Research J.*, **30**, 543 (1960).—A device for abrading fibers by rotating the fiber between two pieces of ground glass covered with a fine optical abrasive powder is described. C.L.D.

Hydroxyproline and the Shrinkage Temperature of Collagen. B. J. Rigby and J. D. Spikes. *Nature*, **187**, 150–51 (1960).—Data of previous investigators on the shrinkage temperature and hydroxyproline content of human dermal collagen are shown to follow the Gustavson rule on the relation between shrinkage temperature and hydroxyproline content. Some speculations about the biological implications of a below-normal hydroxyproline content in human collagenous structures are made. The low values of the hydroxyproline content of fibrinoid tissue from rheumatic fever patients and in rheumatoid arthritis would give the collagen a shrinkage temperature of 40°C.: the rheumatic effects may be the result of structural breakdown of the collagen at body temperatures close to the shrinkage temperature. The high temperatures associated with rheumatic fever may be more damaging to young patients than to adults because of the fact that the hydroxyproline content may increase with age. C.L.D.

States of Aggregation of High-molecular Compounds. IV. Collagen, Gelatin, and Silk Fibroin. R. I. Fel'dman. *Kolloid. zhur.*, **21**, 499–506 (1959); abstr. in *J. Soc. Dyers Colourists*, **76**, 466 (1960).—The method of linear dilatometry already developed can be employed successfully when studying the behavior of fibrous proteins, e.g., temperature-dependent changes of length and weight for collagen (1°–252.5°C.), gelatin (15°–90°C.), and silk fibroin (2°–225°C.). Of the complex factors affecting linear stretching and contraction of protein an important role is assigned to changes in moisture content. In fibrous proteins part of the adsorbed water is less strongly bonded than in polyamides. Changes in the system around 40°C., as shown in dilatometric curves, are connected with very high mobility on the part of some adsorbed water and confirm the presence of hydrogen bonding. A different, more intense bonding is indicated by transition points near 100°C.; changes in dilatometric curves near 190°C. may be caused by the protein component becoming highly elastic. It is emphasized that the point of softening of dry collagen (approximately 240°C.) and of silk fibroin is close to the analogous point for synthetic polyamides. Changes in length are given in relative units rendering comparison on an absolute scale impossible. C.L.D.

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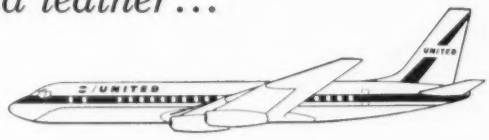

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Leather Buyer Explains Why Suedes Fatliquored With Synova Make A Hit With The Fashion World

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